



Morphology of single inhalable particle inside public transit biodiesel fueled bus

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Abstract

In an urban-transit bus, fueled by biodiesel in Toledo, Ohio, single inhalable particle samples in October 2008 were collected and detected by scanning electron microscopy and energy dispersive X-ray spectrometry (SEM/EDS). Particle size analysis found bimodal distribution at 0.2 and 0.5 μm . The particle morphology was characterized by 14 different shape clusters: square, pentagon, hexagon, heptagon, octagon, nonagon, decagon, agglomerate, sphere, triangle, oblong, strip, line or stick, and unknown, by quantitative order. The square particles were common in the samples. Round and triangle particles are more, and pentagon, hexagon, heptagon, octagon, nonagon, decagon, strip, line or sticks are less. Agglomerate particles were found in abundance. The surface of most particles was coarse with a fractal edge that can provide a suitable chemical reaction bed in the polluted atmospheric environment. The three sorts of surface patterns of squares were smooth, semi-smooth, and coarse. The three sorts of square surface patterns represented the morphological characteristics of single inhalable particles in the air inside the bus in Toledo. The size and shape distribution results were compared to those obtained for a bus using ultra low sulfur diesel.

Key words: individual particle; environmental scanning electron microscope; urban-public transit bus; ultra low sulfur diesel

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Introduction

For last 40 years, air pollution has been the main focus of attention in the United States cities. Suspended fine particulate matter is considered as the criteria pollutant by the National Ambient Air Quality Standards (NAAQS) promulgated in July 1997 (Koren, 1995). Particulate health studies, coupled with the new NAAQS, have generated increased interest in analytical techniques capable of measuring the size, shape, morphology, and chemical composition of individual aerosol particles. Many studies reporting size, morphology, and chemical composition of individual aerosol particles have been conducted. However, particle shape analysis is a relatively new field of investigation (e.g., Colbeck et al., 1997; Shandilya, 2002; Shandilya and Kumar, 2009). In addition, studies of indoor particulate pollution have been carried out in some big cities in the US for many years (Samet, 1993; Kadiyala and Kumar, 2008a, 2008b; Vijayan et al., 2007; Vijayan and Kumar, 2008a, 2008b; Shandilya and Kumar, 2009).

Aerosols produced locally can influence far-reaching areas by transportation mechanisms (Prospero and Savoie, 1989). There is evidence that exposure to particulate matter (PM) from traffic and other combustion sources impact human health more severely than PM (Laden et al., 2000;

Shandilya, 2002; Shandilya et al., 2006, 2007, 2009; Shandilya and Kumar, 2009; Vijayan and Kumar, 2008; Nerella and Kumar, 2009), implying that the health effects associated with PM would be related mostly to anthropogenic emission sources (Kunzli et al., 2000; Schreiner et al., 2006; Shandilya et al., 2009). Kadiyala and Kumar (2008a, 2008b) reported that the various factors affecting indoor air quality for the bus used in this study are indoor sources of pollutants (people, furniture, etc.), ventilation, outdoor air quality, meteorology, pollutant decay, and vehicular traffic. The factors influencing the indoor pollutant levels keep changing randomly as the bus is in motion.

The morphology of atmospheric particles received significant importance in recent years due to the effect of the particles' shape on their radiative and chemical properties. Wang et al. (2003) and Kalashnikova and Sokolik (2002) studied the role of the non-spherical shape of dust particles on their optical properties. Moreover, the role of the shape of particles is sufficiently large as to affect the retrievals of aerosol optical properties from satellite and ground-based remote sensing observations.

As the technique is nondestructive, particles of interest can be relocated for further analysis. Single particulate of PM_{10} has been widely studied in the USA (Kim and Hopke, 1988; Shandilya and Kumar, 2009b). Computer controlled scanning electron microscopy is used by the

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US Environmental Protection Agency to determine particle size, shape, and elemental composition of particles. Micrographs of an individual particle can be obtained to provide particle morphological data. Chemical and physical characterization of individual particles can potentially reveal source information that cannot be determined through bulk chemical characterization, such as atomic absorption spectroscopy and is, therefore, complementary to bulk elemental analysis techniques.

An understanding of the physical and chemical characteristics of PM is needed to establish the relationship between of emission sources of public transport vehicles and indoor PM concentrations. In this way, the classification is based on the morphological characteristics of the aerosol, leading to a better qualitative description of the suspended particles. The shape of a particle collected inside the urban-transit bus, fueled by biodiesel, has only been reported by Shandilya and Kumar (2009b). This study showed that these data contribute to the understanding of the sources of a subset of particulate matter with potentially significant health consequences in an indoor bus microenvironment.

1 Materials and methods

1.1 Sampling

The particulate matter sample was collected inside the urban transit bus operated by the Toledo Area Regional Transit Authority in Toledo on Route 20, as shown in Fig. 1.

A Grimm dust monitor 1.108 was used under 1.2 L/min with the size range of 0.3–20 μm (aerodynamic diameter) using 47 mm PTFE filters (Grimm Aerosol Technik GmbH & Co., USA) with 0.3 μm pores for the microscopic analysis. One collective daily sample from October 2, 2008 to October 31, 2008, for 453 hr was collected for the period of an ordinary day's condition inside an HVAC-equipped biodiesel-fueled bus. Another one collective daily sample

(from October 3, 2008 to October 31, 2008 for 475 hr) was collected for the period of an ordinary day's condition inside an HVAC-equipped ULSD-fueled bus. All the above samplings were conducted in a homogeneous environment inside the bus and sampling was continuous (except some breaks) using the same filter. The collected samples were stored in a deep freezer before analysis.

1.2 SEM/EDS detection

The whole filter paper was mounted on a smooth Cu-alloy stub for the scanning electron microscope (SEM) analysis. The instrument used was a SEM (Quanta 200 3D, FEI/Phillips, Germany), coupled with an Energy Dispersive X-ray Spectrometer (EDAX Genesis, US) to obtain the morphology, size, shape, and chemical composition of individual particles. Elements with atomic numbers less than 11, and the super-tiny particulates less than 1 μm , are not considered during visual examination. During the SEM/EDS detection, on each sample, around 100 particles were scanned and observed. The SEM/EDS was not able to clearly resolve less than 1 μm , therefore, all the results are rounded off to nearest whole number. SEM/EDS working parameters are shown in Table 1.

Table 1 SEM/EDX parameters used for the analysis of the two filters

SEM		EDX	
High tension (kV)	20	Image resolution	512 × 400
Pressure (Pa)	119.98	Particle size (μm)	1–20
Spot size	4	Acquisition time (sec)	10 (live)
Working distance (mm)	10		

1.3 Particle size and shape detection

The particle size and shape analyzer (Particle Analysis Tab, Genesis Software, US) was used. Roughly 500 to 1000 particles need to be characterized to get a representative sample (Mamane et al., 2000), depending on the sample complexity and the overall research objectives.

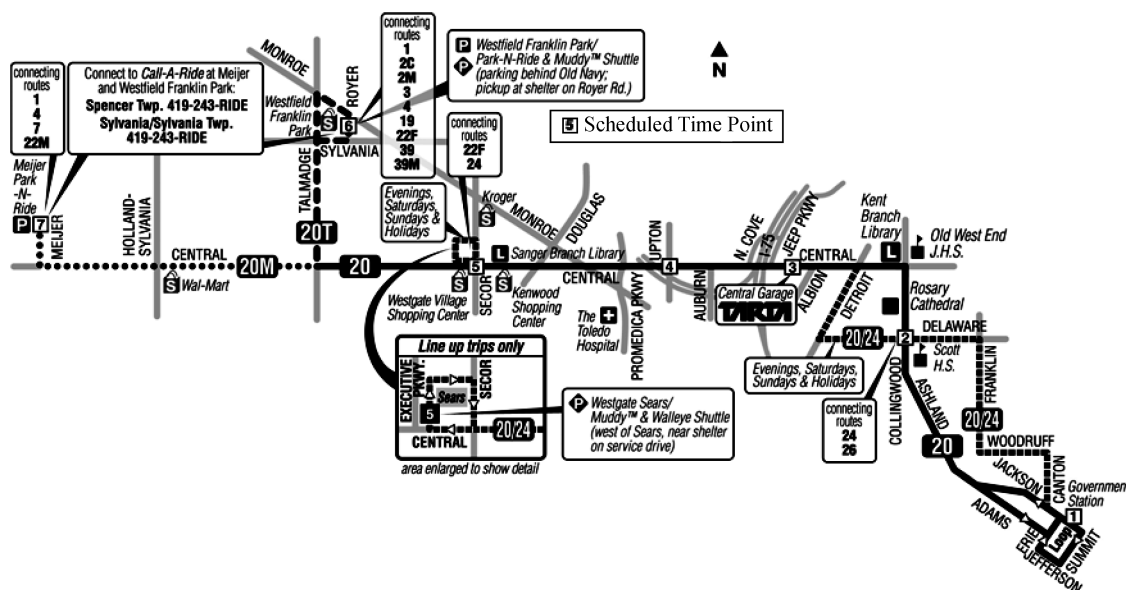


Fig. 1 Map showing route 20 (<http://www.tartabiodiesel.org/>, accessed from 2007 onwards).

In the study, 1884 and 14,021 particles were analyzed using automated X-ray and morphology data from discrete grayscale features in a sample collected in biodiesel- and ultra low sulfur diesel (ULSD)-fueled buses, respectively. To be analyzed accurately, it was made sure that the particles had grayscale contrast from the background and were reasonably well separated from each other. In order to avoid any edge effect, charging, and variable background intensity, the analysis was conducted in the environmental mode. The image collected in an environmental mode showed a better contrast between particles and background, and gray level thresholds were calibrated for consistency and reproducibility. The resolution was chosen based on the size of the particles and the magnification was applied for the analysis. The optimization of gray level by the alignment of noise level near to mid-axis, by adjusting brightness and contrast, is shown in Fig. 3.

A line chart showing the size distribution was drawn. The counting and size distribution analysis was carried out on randomly selected observation fields. All particles within a field of view were counted with their diameter and area measured with the help of Genesis Software in real time with EDX. Tightly bound agglomerates were considered and counted as single particles. Secondary electron images for each and every particle and analysis field were obtained. The magnification for the single particle images was adjusted based on particle size. The secondary and X-ray signals were collected in synchronization with the position of electron beam to provide the highly detailed spatial and compositional information of microscopic features.

2 Results and discussion

2.1 Particle size analysis

The size analysis was based on the images with most particles within the smallest size range (between 0.07 and 0.5 μm) for the biodiesel-fueled bus, which is very different from particle size distribution for particles collected inside the ULSD-fueled bus. For larger particles, the number concentration was less and only a small number of particles with diameters greater than 1.8 μm were found for biodiesel-fueled bus, while the majority of particles found in ULSD-fueled bus were greater than 8.9 μm . After

compilation in a spreadsheet, the size data (diameter) are categorized in Fig. 2.

The large proportion of all particles was found to be in the sub 0.5 μm diameter category for the biodiesel fueled bus. However, the large portion of particles for the ULSD-fueled bus was in 8.9 μm diameter category. The peaks for bi-modal size distribution were found at 0.2 and 0.5 μm for the biodiesel fueled bus. The peaks for multi-modal size distribution were at 8.9, 9.9, 10.9, 11.7, 12.5, 13.3, 14, 14.7, 15.3, 15.9, 16.5, 17.1, 17.7, 18.2, 18.8, 19.3, and 19.8 μm , respectively, for the ULSD-fueled bus. No diameter particle greater than 17.5 μm and 19.8 μm was found in the samples collected in both the buses. This suggests that the Grimm sampler worked properly with a cutoff diameter of 20 μm . The bi-modal distribution observed during this study for the biodiesel-fueled bus is similar to the one reported by Shandilya and Kumar (2009b). The particle size distribution spans very narrow in October with more fine mean size value. This observation indicates the absence of ground alteration emission for biodiesel fueled bus but presence for ULSD-fueled bus.

2.2 Particle shape sorts

By examining the species microstructure, it was concluded that regular square and round symmetries were the prevailing structures. The particles differed in both morphology and size and were in single and clustered forms. The projections shown in Fig. 3 are commonly seen when a single particle lies flat in the specimen holder, which is not tilted in the SEM image. One important deficiency of the SEM characterization is the inability to explore the three-dimensional morphology of the particles and thus assume that the investigated particles on the substrates are flat surface.

After compilation in a spreadsheet, the shape data (shape factor) were categorized and plotted as in a histogram. The major proportion of all particles was found to be in the square shape category. As established by many authors (Casuccio et al., 1983), the SEM method gives an intuitive way to identify a particular matter by its outlook. The crystalline nature of the particle's morphology suggests that it may have formed during post sampling from interaction with atmospheric gases, such as sulfur-di-oxide.

Based on the images of particle surface shape gained by

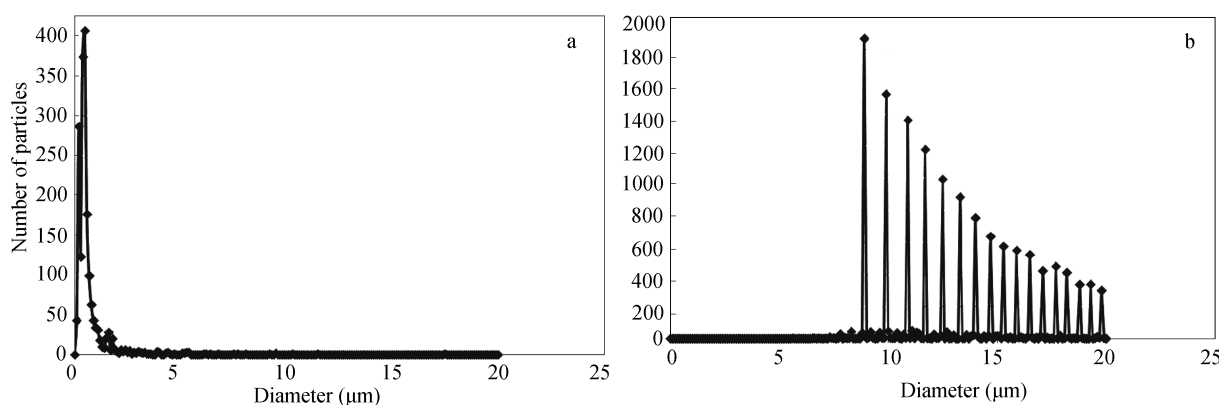


Fig. 2 Particle size distributions. (a) inside bio-diesel fueled bus; (b) inside ULSD-fueled bus.

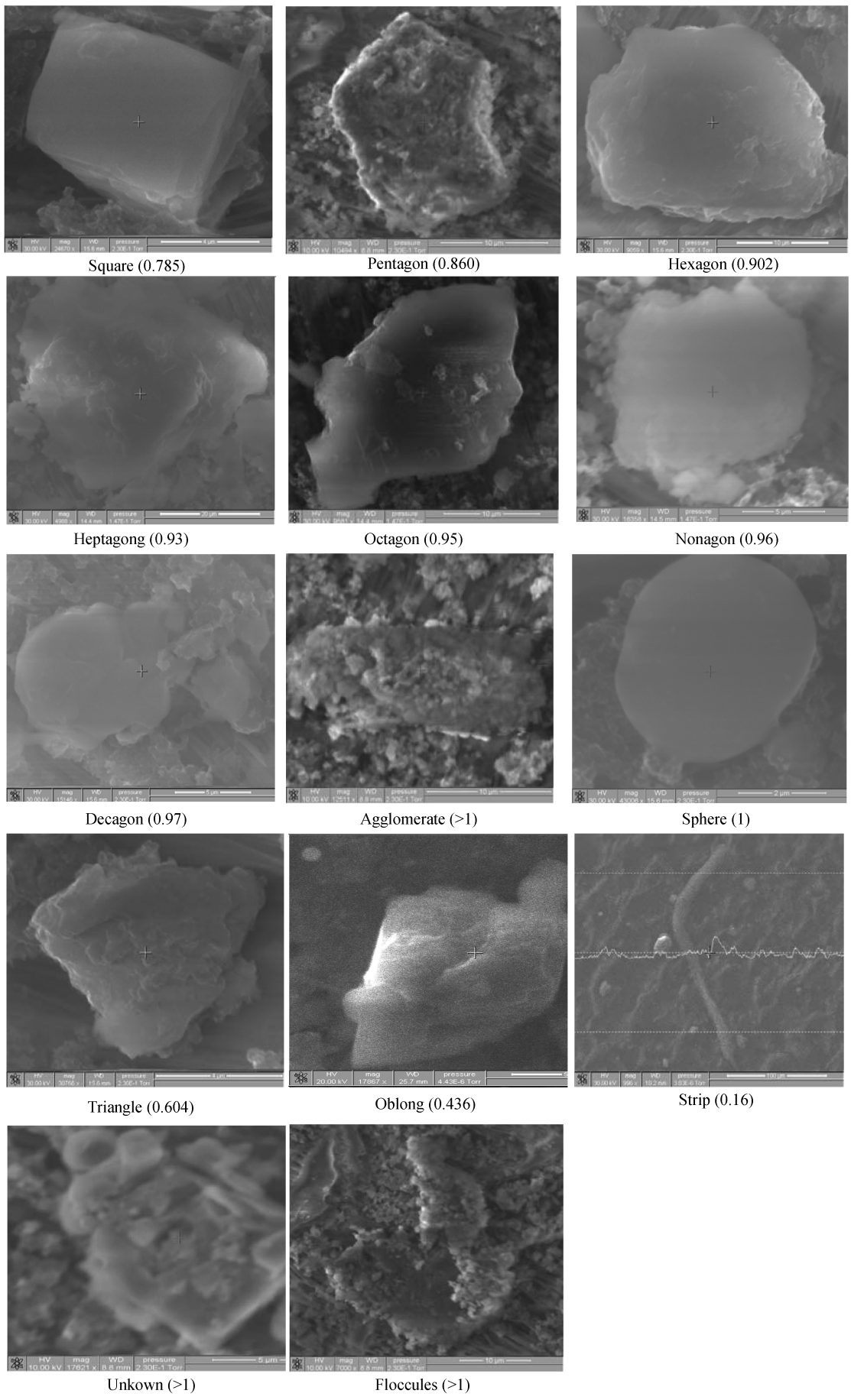


Fig. 3 Different particle shapes. Number shown in parentheses are shape factors.

the SEM/EDS analysis, 14 clusters of particulate morphology of PM, inside the urban transit bus in Toledo, have been sorted out (Fig. 3).

2.2.1 Particle shape distribution

The particle shape factor (S_F) is given by Eq. (1):

$$S_F = \frac{4\pi A}{P^2} \quad (1)$$

where, A is the particle area and P is the particle perimeter, A and P can be expressed as following Eqs. (2) and (3) respectively.

$$A = \frac{S^2}{4 \tan \frac{\pi}{N}} \quad (2)$$

$$P = N \times S \quad (3)$$

where, N is the number of sides in the polygon and S is the side of the polygon. Shape factors for different shapes are listed in Fig. 3. The major limitation of our study is grouping agglomerate, floccules, and unknown particles together and assigning them collectively into a shape factor greater than 1.

Figure 4 shows the particle shape distribution. For the sample collected inside the biodiesel-fueled bus, the majority of particles were found in square, round, oblong, and triangle shapes, followed by strip, pentagon, hexagon,

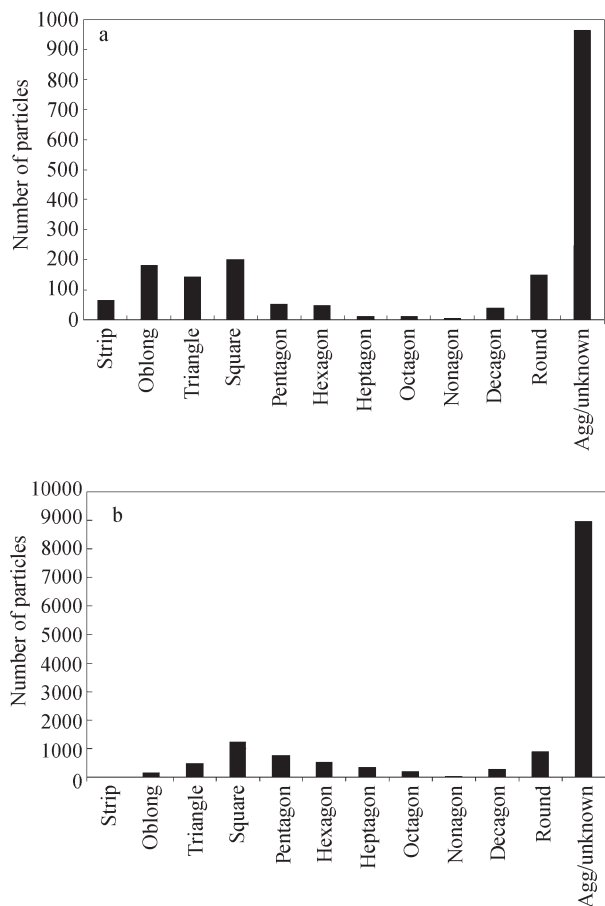


Fig. 4 Particle shape distribution. (a) inside biodiesel-fueled bus; (b) inside ULSD-fueled bus.

and decagon. The highest number of particles falls in the category of agglomerate, unknown, and floccules. Whereas, the majority of particles were found in square, round, pentagon, triangle and hexagon, followed by heptagon, octagon, decagon, nonagon and strip, in the sample collected inside the ULSD-fueled bus. Highest particles fall in the category of agglomerate, unknown, and floccules. The comparison of particle shape distribution of samples collected inside biodiesel fueled bus, and the ULSD-fueled bus, is similar in nature. However, the magnitude of different shape particles differs.

#	Spectra	Dia. (μm)	Element
a		3	Na, Cl
b		9	Al, Si, Mo
c		5	Al, Mo, Cl, Pd
d		7	Al, Si, Mo, Ca
e		3	Na, As, Al, Si, Cl, K
f		7	Na, Si, S
g		5	Al, Mo, Cl, Pd
h		2	Zn, Al, Si, Mo
i		6	Na, Si, Mo
j		10	Na, S

Fig. 5 Different shaped particle, diameter and chemical spectra. (a) smooth surface square particle; (b) semi-coarse square particle; (c) coarse and holed square particle; (d) pentagon particle; (e) Hexagon particle; (f) agglomerate particle; (g) sphere particle; (h) oblong particle; (i) Triangular particle; (j) unknown shape particle.

2.2.2 Square

Irregular shaped square grains (Fig. 5a) are quantitatively high in the samples detected in the urban transit bus in Toledo. There are three impressed surface patterns of the square particles inside the urban transit bus in Toledo: smooth (Fig. 5a), semi-coarse (Fig. 5b), and coarse (Fig. 5c). Each pattern corresponds to different elemental components: the smooth square (size about 3 μm) to Na, Cl-dominant; the semi-coarse (size about 5 μm) to Al, Si-dominant; and the coarse to Mo-dominant, especially when the squares are captured separately. The squares show the presence of other elements that illustrate they conglutinated. The smooth square particles were found to contain Na, Mo, Cl, Ca, Al, Si, S, K, Pd, Mg, Ag, Ti, V and Fe. The semi-coarse square particles were found to have Na, Mo, Pd, Cl, Al, Si, S, Cl, K, and Ca. The coarse square particles were found to have Al, Mo, Cl, Pd, Na, Mg, and Ca. Some irregular diamonds have rather smooth and flat surfaces with obvious Ca peak in the elemental spectrum. The amount of particles with smooth surfaces is relatively more than the coarse particles.

2.2.3 Pentagon

Irregular shaped pentagon particles (Fig. 5d) are also present in the samples detected in the urban transit bus in Toledo. Most of the pentagon particles had coarse surface and irregular shapes, which indicates the presence of Al, Si, Mo, Ca, and Mg. The surface of these particles was coarse and holed, with the average diameter of 5 μm .

2.2.4 Hexagon

Irregular shaped hexagon particles (Fig. 5e) were almost equal to pentagonal particles in the samples detected in the urban transit bus in Toledo. Most of the hexagonal particles were having coarse surfaces showing the presence of Na, As, Al, Si, Cl, and K. The surfaces of these particles were coarse and smooth, with an average diameter of 6 μm .

2.2.5 Agglomerate

Agglomerate shaped particles (Fig. 5f) are less quantitative than the irregular diamond, but are a little larger in size. Element components of agglomerate particles are quite similar to those of the irregular square particles but with a different X-ray counts rate. In some particle surfaces of agglomerates, elements Na, Si, and S are measured by EDS. These agglomerate particles are actually the combination of more than two particles.

2.2.6 Sphere

Sphere particles (Fig. 5g) are generally smaller than square particle with an average diameter under 5 μm . Spherical particles are considered as geometrically round. The round particles were found to have Al, Mo, Cl, and Pd. The particle surface was coarse and holed.

2.2.7 Column or stick

These particles are shaped long and conical. This sort of grain usually bears some external physiological texture, and can be found in samples inside the urban transit bus in Toledo. The X-ray detecting counts for these particles are

quite low, which demonstrates that they are biomass.

2.2.8 Oblong

Shaped long and triangular, this sort of grain (Fig. 5h) usually bears some physiological texture externally, and can be found in samples inside the urban transit bus in Toledo. These were found to contain Na, Si, Mo, In, Ca, Mg, Fe, Zn, and Al.

2.2.9 Triangle

Triangular particles (Fig. 5i) were also found on the filter. The elements found were Na, Mg, Al, Si, Mo, Cl, K, Ca, Ti, V, Fe, and S. The average diameter was 5 μm .

2.2.10 Unknown

Some deformed or nondescript particles (Fig. 5J) just account for the minority in the samples with elemental spectrum peaks of Na and S, which may be explored further.

2.3 Sources of particulate matter

Based on the morphological features, it can be assured that the irregular diamond particles are derived from soil and surface geological deposit as the product of mechanical abrasion (Kaegi, 2004). The agglomerate and spherical particles are produced from the combustion of coal (Ramesh and Koziski, 1999), while the floccule particles are from the discharge of vehicles (Colbeck et al., 1997), and the column or stick shaped particles are from bioactivities (Crook and Sherwood-Higham, 1997). In Toledo, a distinct environmental problem is the fugitive dust derived from bare soil, construction activities, vehicle discharge, industry emission, and street deposit. Due to repeated depositing, accumulating, and re-suspending processes, the particles from the above sources mix and create dust, which is a serious nuisance. The detection of PM particles by ambient sampling represents intermingling of each original discharge source and re-suspension of street dust. The ratio of particle number percent by calculation under SEM can only show source categories.

2.4 Chemical composition

Elements found under the microscope are Na, Mg, Al, Si, Mo, Ca, Cl, K, Ti, V, Fe, Pd, S, As, Ag, In, and Zn. The possible sources are listed in Table 2. The strong correlation of sodium and chlorine suggests that chloride is converting to sodium chloride in the atmosphere. The major source of smooth and shiny square particles is the salt that usually is put on the road and on sidewalks to prevent skidding. Table 2 lists the possible sources contributing to the particles found inside the urban-transit bus in Toledo. Based upon the prevalence of square shaped particles, it can be concluded that major sources contributing to the aerosol levels inside the public transit bus include road salt, lake bed, road dust, soil, vegetative burning, incinerator, oil fired power plant, smelter fine, motor vehicle, construction, coal fired boiler, and road traffic background.

The particle shape distribution of particles collected is identical for the first three dominant shapes in both the buses. It possibly suggests that similar sources are

Table 2 Elements and their possible sources

Element	Possible sources
Na	Road salt, lake bed, paved and unpaved road dust, agriculture and natural soil, vegetative burning, incinerator, oil-fired power plant, smelter fine.
Mg	Natural soil, smelter fine.
Al	Motor vehicle, incinerator, paved and unpaved road dust, construction, agriculture and natural soil, lake bed, oil fired power plant, coal fired boiler.
Si	Motor vehicle, incinerator, paved and unpaved road dust, agriculture and natural soil, lake bed, oil fired power plant, coal fired boiler, construction.
Mo	Secondary/industrial sources, resuspension, salt, diesel vehicles, road traffic background (Harrison et al., 2003).
Ca	Motor vehicle, paved and unpaved road dust, construction, agriculture and natural soil, lake bed, incinerator, smelter fine, coal fired boiler, oil fired power plant, vegetative burning.
Cl	Incinerator, motor vehicle, lake bed, agriculture and natural soil, paved and unpaved road dust, vegetative burning, coal fired boiler.
K	Paved and unpaved road dust, construction, agriculture and natural soil, lake bed, incinerator, coal fired boiler, oil fired power plant, smelter fine, vegetative burning.
Ti	Paved and unpaved road dust, construction, agriculture and natural soil, lake bed, incinerator, smelter fine, coal fired boiler.
V	Coal fired boiler, smelter, oil fired power plant, and incinerator.
Fe	Vegetative burning, motor vehicle, paved and unpaved road dust, construction, agriculture and natural soil, lake bed, incinerator, coal fired boiler, smelter fine.
Pd	Auto catalyst, incinerator.
S	Paved and unpaved road dust, construction, agriculture and natural soil, vegetative burning, lake bed, motor vehicle, incinerator, coal fired boiler, oil fired power plant, smelter fine.
As	Coal fired boiler, oil fired power plant, smelter fine.
Ag	Incinerator.
In	Smelter.
Zn	Paved and unpaved road dust, construction, agriculture and natural soil, vegetative burning, motor vehicle, incinerator, coal fired boiler, oil fired power plant, smelter fine.

contributing to particulate levels inside the buses. However, interestingly particle size distribution has very distinct features for the particulates collected in both the buses. This possibly suggests differences in particulate chemical composition and source origin.

2.5 Particle surface characteristics and possible atmospheric reaction

Airborne particles play an important catalytic role in the atmospheric chemical reaction (Boke et al., 1999), and the particle surface character is influential (Dall'Osto and Harrison, 2006). In the indoor environment, only a few particles possess a smooth surface and most of them are fractal. With the enlarged surface area by the cracked and holed process, these fractal particles can provide a suitable environment and medium for the secondary atmospheric reaction.

3 Conclusions

In the typical indoor air of an urban transit bus fueled by biodiesel in Toledo, single inhalable particles' morphology was characterized by the 14-shape clusters by quantitative order: triangle, square, pentagon, hexagon, heptagon, octagon, nonagon, decagon, agglomerate, sphere, floccules, column or stick, and unknown. The square particles are common in all kinds of samples; sphere particles are more, and column or stick are less; agglomerate and floccules particles are found but not differentiated in this research. The comparison of particle shape distribution of samples collected inside a biodiesel-fueled bus and ULSD-fueled bus is quite similar except strip particles were lower in samples collected inside the ULSD-fueled bus. The surface of most particles is coarse with a fractal edge, which can provide a suitable chemical reaction bed in the polluted atmospheric environment. The three sorts of surface patterns of squares are smooth, semi-coarse, and coarse, and correspond to the elements of Na, Cl-dominant, Al, Si-dominant, and Mo-dominant. The soot particles are present in the wrapped or captured form with other fine particles. This mixed process can compound particle components to enhance their potential toxicity, but to increase their deposition velocity by possible increased weight. The particle shape distribution of particles collected is identical in both the buses for the first three dominant species. The particle size distribution collected under the microscope is totally different for the biodiesel and ULSD-fueled buses. This possibly suggests that the particles may be of different composition and are coming from different sources. This point needs major investigation. The three sorts of square surface patterns represent the single inhalable particle's morphology characteristics inside the urban transit bus fueled by biodiesel in Toledo. They are different from those in ambient air, and deserve further research.

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